## **ABSTRACT**

A process for the recovery of acrylonitrile or methacrylonitrile from an aqueous solution comprising subjecting the said solution to a water extractive distillation by feeding the solution to a distillation column and performing said extractive distillation and using solvent water introduced at the top of said column, removing a first overhead vapor stream of acrylonitrile or methacrylonitrile with some water from the top of the column, and a first liquid stream containing water and impurities from the bottom of the column, the contents of said column maintained at a substantially neutral pH by adding a sufficient amount of at least one alkaline compound selected from ammonium carbonate, ammonium bicarbonate, ammonium carbamate, and alkylene diamines to the overhead decanter and/or to the solvent water.

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